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(71) Applicant
Matsushita Electric Works Ltd., (Japan),
1048 Oaza Kadoma, Kadoma-shi, Osaka, Japan

(72) Inventors
Tamotsu Akasaka, Kenichi Matsui,
Takashi Soda, Yasushi Sawada
Takeru Murakami,

(74) Agent and/or address for service
Page White & Farrer, 5 Plough Place, New Fetter Lane,
London, EC4A 1HY

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(54) Producing Inorganic Hardened Compositions

(57) A method for producing asbestos-free cement boards, such as by a scoop-sieving process to obtain inorganic hardening compositions for curing utilises a cement slurry which includes fibrillated wood pulp having specified characteristics optionally unbeaten virgin pulp and if necessary, filler and reinforcing fibers for obtaining 4 to 15 wt.% of the total solid in concentration and below 5 cm^4/sec for filtration coefficient for the slurry. The amounts of the fibrillated pulp and the virgin pulp are 1 to 5 wt. % and 0.5 to 1.0 wt. % of the total solid content, respectively. The pulp is bleached or unbleached wood pulp from conifer and/or broadleaf tree. The filler is sepiolite and/or bentonite, and/or crystal or noncrystal silica of below 5 μ in grain size, in an amount of 1 to 10 wt. % of the total solid content. The reinforcing fibers are vinylon and or acrylic fibers having specified characteristics and being present in an amount of 0.3 to 2 wt. % of the total solid content, or wollastonite in the amount of 2 to 15 wt. % of the total solid content.

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SPECIFICATION

Method for Producing Inorganic Hardened Compositions

The present invention relates to a method for producing inorganic hardened compositions, or boards, which may be used as architectural materials, and particularly to a method for producing inorganic compositions such as cement-based inorganic architectural materials without using asbestos.

The inorganic hardened compositions containing cement as a binding agent and asbestos as a reinforcing agent are used extensively. Asbestos is used because it enhances the reinforcing effect on the inorganic compositions remarkably. The asbestos also makes it possible to obtain the inorganic hardened composition by use of a scoop-sieving production method, such as the known Hatchek system, that is suitable for mass-production. In this method, the slurry containing raw materials is processed by a scopping machine such as the Hatchek processing machine. The basic formation obtained by such a process is cured in order to obtain the inorganic hardened composition. This production process becomes practicable when the solid content of the asbestos exceeds 5 wt. %.

However, the use of asbestos may cause environmental pollution, and the continued future use of asbestos will create serious public health problems.

Because of such environmental problems, studies have been made to produce inorganic hardened compositions without asbestos. One example is an inorganic hardened composition which contains pulp in place of asbestos, and such products have already been marketed.

However, this inorganic hardened composition is defective and inappropriate for general use as a building material because it is inflammable. For producing this inorganic hardened composition by the scoop-sieve production method the solid content of the raw pulp must equal or exceed about 6 wt. % (% by weight). However, when such a large concentration of pulp is used, the resulting inorganic hardening composition becomes combustible. In addition, this inorganic hardening composition has the disadvantage that it is not strong enough particularly when it absorbs water. Hence, such a composition is not suitable as a building material for facings.

Presently, studies are being made to substitute fibers for asbestos (besides pulp), various inorganic fibers such as glass fiber, steel fiber, carbon fiber, and wollastonite, and various organic fibers such as vinylon, acryl, polyethylene. However, such fibers are all thicker than asbestos and have a low affinity for cement. Therefore, none of them has reached the stage to be used by itself as single fiber material.

A method for producing asbestos-free cement boards with cotton of a solid content 2—25% obtained by adjusting the beating degree (or schopper freeness (20—80° SR is disclosed in Japanese Patent Application Laid-Open No. 1981-114857. However, since cotton has long fibers, it is very difficult to make a dispersion in the slurry; although the reinforcing effect can be found if the dispersion is carried out satisfactorily.

When the freeness is increased by cutting the cotton fibers short, the dispersibility is improved. However, the reinforcing effect of the cotton fibers is quite low. Furthermore, the cotton fibers cost about twice as much as wood pulp.

The present invention provides a method for producing an inorganic hardened composition including the step of curing a composition which is obtained from a slurry containing cement by using a production process such as for example scopping-sieving, said slurry comprising: pulp accounting for 1—5 wt. % (% by weight) of the total solid content of the slurry, 10 wt. % or less of said pulp being of 177 μ or shorter in fiber length and being adjusted with its Schopper freeness at 70°SR or higher through fibrillation; and filler having a swelling degree of more than three times and reinforcing fibers upon necessity, in order to adjust the concentration to be 4—15 wt. % and the filtration coefficient to be below 5 cm^4/sec for said slurry.

The present invention further provides a method for producing an inorganic hardened composition including the step of curing a composition which is obtained from a slurry containing cement using a production method such as, for example, scoop-sieving, said slurry comprising: wood pulp accounting for 1—5 wt. % of the total solid content of the slurry, 60 wt. % or more of said wood pulp being of 590 μ (28 mesh) or longer in fiber length and adjusted with its Schopper freeness at 40—95°SR, unbeaten wood pulp of below 40°SR in Schopper freeness, said unbeaten wood pulp accounting for 0.5—1.0 wt. % of the total solid content of the slurry, and filler and reinforcing fibers, if necessary, to be compounded in order to adjust the concentration to be 4—15 wt. % and the filtration coefficient to be below 5 cm^4/sec for said slurry.

Embodiments of the present invention will now be described by way of example only.

After conducting many studies, the inventors have found that nonflammable and extremely strong hardening compositions can be mass produced by using a process in which the pulp is fibrillated by beating without being shortened, in combination with virgin pulp that is not fibrillated but merely processed by disaggregation.

The present invention was completed based upon that finding.

In keeping with the principle of the invention, the above objects are accomplished by a unique method for producing inorganic hardened compositions. In the preferred method is used pulp of 177 μ

in length to which is applied fibrillation while keeping it below 10 wt. % of the total pulp amount in order to set the Schopper freeness at 70°SR or higher.

The content of the pulp thus obtained in the slurry is kept at 1—5 wt. % of the total solid content of the slurry. If necessary, a filler with a degree of swelling of more than three times and the reinforcing fibers are compounded in addition to the pulp. The slurry is then adjusted to be 4—15 wt. % in concentration and less than 5 cm^4/sec in coefficient of filtration.

Also used in the invention is a pulp having a fiber length of more than 590 μ (28 mesh). The pulp is maintained to be present in a composition ratio of more than 60 wt. % compared with the total content of the pulp obtained. The pulp resulting is adjusted to be 40—95°SR in Schopper freeness (degree of water filtration). Then the pulp is compounded with the virgin pulp that is processed only with disaggregation This virgin pulp is of less than 40°SR in Schopper freeness. The ratio for compounding is 1—5 wt. % for the pulp and 0.5—1.0 wt. % for the virgin pulp against the total content of the solid components. With such compounded pulp, even with less than 6 wt. % in amount, the production or scooping by the Hatchek system is feasible. In this case, the virgin pulp is effective in bringing about improvement in water drainage (filtration) during the processes of a suction making, rolling, and pressing.

In the above, the degree of swelling and the coefficient of filtration is defined as follows:

$$\text{Degree of swelling:} \frac{\text{Amount of Water absorbed (measured) after 24 hours}}{\text{Weight of filler before absorbing water}}$$

Coefficient of filtration: Coefficient per unit filtration area during constant-pressure filtration

$K=2\ V/(d\ \theta/dv)$

V; Volume of filtrate (cm^3)
θ; Filtration time (duration) (sec)
60 mesh wire gauze is used

In the preferred method of the present invention, any type of water-based cement may be used as a binding material without specific restrictions. For example, Portland cement, or Portland blast furnace cement may be used. As the wood pulp, bleached or unbleached kraft pulps of needleleaf trees and broadleaf trees are preferable for use. Used-paper, such as sulfite paper or kraft paper, when used in large quantity, may cause unsatisfactory setting of the cement because of impurities contained in such used-paper. However, in general, since the fiber length of the used-paper is short and Schopper freeness is relatively high, they are frequently used, although in small quantity, together with asbestos. Such used-paper may also be used in the present invention as long as more than 60 wt. % of the total content of the wood pulp containing such papers has a fiber length of 590 μ or longer as mentioned previously and its Schopper freeness is within the range of 40—95°SR.

To attain 40°SR in freeness, two methods are conceivable: pulp cutting or pulp fibrillation with minimum cutting. Pulp cutting is effective in improving the freeness, but is not sufficient to reinforce the hardened cement composition. On the other hand, pulp fibrillation with restricted pulp cutting is effective to increase the freeness while also reinforcing the hardened cement composition. However, the fibrillation must be controlled, or else the pulp cutting also proceeds further. The characteristic point of this invention is that the pulp used is fibrillated but the fibrils cut short in the pulp are limited to be present in the lowest possible concentration.

In one method according to the present invention, pulp made from needle-leaf tree and/or broadleaf trees are used. The Schopper freeness of the pulp is brought to 70°SR or higher. The fibril content with fiber length of less than 177 μ is less than 10 wt. % of the total amount of the pulp. Such pulp are used in an amount of 1—5 wt. % (hereafter, will be abbreviated as %) of the total solid content of the slurry. When the content of the fibrillated pulp is less than 1%, even if the fine grained inorganic filler which is capable of lowering the filterability is added, it is impossible to lower the coefficient of filtration to the point enabling production using the Hatchek system. On the other hand, if the fibrillated pulp exceeds 5% in its content, although production is feasible, when the amount of the other organic reinforcing fibers is taken into account, it becomes impossible for the product to meet the requirement in terms of non-inflammability.

The Schopper freeness of the needle-leaf tree pulp or the broadleaf pulp, when beaten normally, is below 40°SR. If such pulp is used in content of less than 5%, even with the combined use of the fine grained inorganic filler that is capable of lowering the filterability, production using the Hatchek process is impossible. It means that because of the excessively high filterability (i.e., the drainage is too good), the cement grains slip out into the filtrate. Therefore, the presence of the pulp beaten to be above 70°SR in Schopper freeness in an amount of more than 1% is an imperative requirement for producing (by, such as, scoop-filtration) the non-inflammable hardened composition.

Next, as to the fiber length of the fibrillated pulp, the beating is done by beating machines such as PFI mill, single disc refiner, double disc reinfer, and simultaneously with the fibrillation, the process to

cut the fibers shorter proceeds. It is necessary to beat the pulp in order to increase the Schopper freeness. However, when too much beating is given, the fibers are cut into short pieces, becoming incapable of serving as a reinforcer of the hardening composition. For example, the fibers of less than 177 μ in length show almost no reinforcing effect, and they are effective only in increasing the Schopper freeness. Therefore, it is desirable to keep the amount of such short fibers as small as possible.

As mentioned above, in this method a pulp having the fiber length of less than 177 μ is used for below 10% of the total pulp content. The reason is that when the content of such pulp exceeds the level mentioned above, the water absorption ratio of the hardened composition increases, resulting in a substantial decrease in strength when it absorbs the water.

Further, in addition to the fibrillated pulp having the Schopper freeness of above 70°SR and less than 177 μ in length counting for less than 10% of the total content of the pulp, a pulp of below 70°SR in Schopper freeness (needle-leaf tree virgin pulp, broadleaf tree virgin pulp, used paper, etc.) may be added. In other words, if the fine grained inorganic filler lowering the filterability is used together, it is not necessary to use the above-mentioned fibrillated pulp alone. The other pulp(s) with the Schopper freeness of below 70°SR may also be used in combination within the range wherein the filtration coefficient of the slurry can be adjusted to be less than 5 cm^4/sec.

The ratio for combined use of the pulp of above 70°SR and below 70°SR in Schopper freeness is set 1:4—5:0 in the invention. When the strength and economy are taken into consideration, the range of 1:1—2:1 is preferable.

In other methods according to the present invention, coniferous tree and/or broadleaf tree pulp with a Schopper freeness of 40—95°SR and wherein pulp with fiber length of more than 590 μ accounts for more than 60% of the total pulp content are used. The concentration of such pulps as a percentage of the total solids is 1—5 wt. % hereafter abbreviated as "%"). Also, needle-leaf tree and/or broadleaf tree pulp which are processed only with disaggregation and which are less than 40°SR in Schopper freeness are used. The concentration of the latter pulp as a percentage of the total slurry solids content is 0.5—1.0%. In other words, if the above-mentioned fibrillated pulp is less than 1% in concentration, even when the filler that is effective in bringing down the filterability is used together with the foregoing pulp, the coefficient of filtration cannot be lowered to the range enabling production by the Hatchek system. Or, even if the coefficient of filtration can be lowered to the range which makes it possible for the production to be accomplished by the Hatcheck process, the amount of the cement grains escaping with the water through the meshes of the net of the cylinder becomes large, and it is impossible to obtain products with expected quality. Furthermore, problems including the clogging of pipes are caused in the production process. On the other hand, when the concentration of the fibrillated pulp mentioned above exceeds 5%, production is feasible, but the product fails to meet the requirement for non-flammability when the amount of the other reinforcing organic fibers is taken into account.

In the case of combined use of virgin pulp of 40°SR or lower in Schopper freeness, a concentration of less than 0.5% still permits the process to proceed with scoop-sieving during production, but trouble occurs during the subsequent process step, i.e., in the dehydration of cake through felt, where the hydroextractability becomes low. As a result, during the process of rolling up the cake onto the making roll, the exceedingly high water content may cause adhesion to the roll surface or failure to maintain shape.

On the other hand, when the virgin pulp concentration exceeds 1%, the coefficient of filtration increases, leading to a high solids concentration in the filtrate, and the yield of cement is brought down to an extremely low level. Yet another disadvantage found in this case is that during the pressure-forming of the clean sheet, the spring-back upon release of pressure increases and the products obtained are all low in specific gravity. As is described previously, for obtaining the fiber-reinforced cement board using the Hatchek system, it is necessary to use the fibrillated pulp in a concentration of 1—5%. However, with fibrillated pulp alone, even though production by scoop-sieving is feasible, there is a drawback that due to the low rate of dehydration through felt, making, rolling and during press work, the products lack sufficient specific gravity.

In the present invention, where 0.5—1% of virgin pulp is included (compared with the method using only the fibrillated pulp), the dehydration of the cake during the production is performed with high efficiency. Accordingly, high specific gravity products, wherein the pulp fibers and cement are homogeneously and intimately mixed can be obtained.

The virgin pulp used in the method according to the present invention is needle-leaf tree pulp or broadleaf tree pulp that is beaten normally and that is below 40°SR in Schopper freeness. When this virgin pulp is used alone, production by the Hatchek method is impractical, even if the other materials such as filler are added. The result is the same even when the amount of the foregoing virgin pulp used is increased to more than 0.5—1% which is the range applied to the method of the present invention. In other words, the filterability is so high that the cement grains slip out into the filtrate. This creates not only inferior physical properties, but also undifferentiated fluid levels. Then production which requires difference in fluid levels is not feasible. Only when the pulp beaten heavily up to 40—95°SR in

Schopper freeness is used in combination with the virgin pulp, can the Hatchek system make high-density products with high efficiency.

Next, the fiber length of the fibrillated pulp is obtained by beating with beating machines such as a PFI mill single disc refiner or double disc refiner. Along with the fibrillation, the process of cutting fibers shorter goes on. It is necessary to beat the pulp in order to make the Schopper freeness higher, but too much beating results in shortened fibers by cutting, thus spoiling the pulp of its effect in reinforcing the hardening products. For example, the fibrillated pulp with fiber length of less than 590 μ shows almost no reinforcing effect but only an increase in Schopper freeness. Therefore, one should limit the amount of such short fiber pulp as much as possible.

In this invention, the reason for setting the amount of pulp with fiber length of 590 μ or longer to be above 60% of the total content of the pulp is that if the content of this pulp is below 60%, the water absorption rate of the hardening composition is increased, and the strength of the hardening composition upon absorbing water is lowered.

As to inorganic filler, those having swelling degree of more than three times are used. For example, inorganic fillers, such as sepiolite, bentonite, which show a high swelling degree when absorbing the water are used. When such filler is used together with the aforementioned pulp, through mixing them with cement and water, the coefficient of filtration of the slurry can be decreased to below 5 cm^4/sec which is the range to obtain the basic formation by using the Hatchek system. By the combined use of the foregoing fibrillated pulp and the expansible inorganic filler, the freeness can be lowered (i.e., the yield of cement is upgraded). It is not clearly known why the freeness is lowered and the yield of cement is increased. However, it is assumed that the swollen inorganic filler is intertwined with the fibe fibers of the fibrillated pulp in satisfactory manner, and they form a reticular structure when they come to be filtrated.

The reason for setting the sweeling degree of the inorganic filler to be more than three times is this: When the inorganic filler with a swelling degree of less than 3 times is used, not much effect was shown for lowering the coefficient of filtration mentioned above. Also, when the amount of the inorganic filler exceeds 5%, there occurs the possibility of lowering the strength (lowering of strength upon absorption of water).

Other filler, having more than three times in degree of swelling used in the invention, is crystal or noncrystal silica of less than 5 μ in average grain size may be used. When such filler is added to the above-mentioned pulp and mixed with cement and water, the coefficient of filtration of the slurry is further lowered, and the slurry is ready to be processed for production. The filler makes it possible to obtain a coefficient of filtration in a range that is practical for production even if beating of the pulp has been limited. This saves power for pulp beating and also is effective in bringing about product flexibility. The composition of materials can be varied not only by the pulp, but also by the filler, depending on the use of the product. Another advantage is that when crystal or noncrystal silica of less than 5 μ in grain size is used as filler, it reacts with the cement during the curing, straightening the product further.

As reinforcing fibers, in addition to the pulp, inorganic fibers such as glass fiber, carbon fiber, steel fiber, wollastonite; organic fibers such as vinylon, acryl, polyethylene, may be used. As organic fiber, vinylon is most preferable, while as inorganic fiber, wollastonite is preferred. Among the vinylon fibers, those partially uneven are desirable. It is common knowledge that of the organic fibers vinylon fiber is the highest in effect of combining with cement because of its hydrophilic group, producing outstanding reinforcement. Combined use of this fiber with the fibrillated pulp and virgin pulp, results in a further improvement in strength. The postulated reason for the above is that the vinylon used along has a small affinity for cement and tends to slip off. However, when it is used together with the foregoing pulps, the vinylon fiber is intimately entwined with the fibrillated pulp as well as with the virgin pulp, which prevents slipping off. Furthermore, if the vinylon used has a partially uneven surface, as a result of heat treatment, etc. during spinning or after spinning, the tendency against slipping off is further enhanced.

As to the vinylon fibers, a thickness of 5—50 μm and a length of 3—10 mm are the most preferable ranges. The preferred content of vinylon fiber is 0.3—3%. When the content exceeds 2% in the ordinary slurry production system, it becomes difficult to effect homogeneous dispersion of the fibers, resulting in a loss of strength. On the other hand, when the content is below 0.3%, the reinforcing effect becomes insufficient. Particularly, in the unhardened state, the ability to maintain the shape becomes poor.

For wollastonite, no particular limit is imposed on the length, thickness, and shape of its fiber, but it is naturally desirable that the wollastonite have as high as possible aspect ratio. The preferred content of wollastonite is in the range of 2—15%. When the content exceeds 15%, the reinforcing effect itself is not lowered, but lowered the specific gravity of the hardening composition reduces the reinforcing effect as a whole.

The slurry is prepared by mixing the above-mentioned starting materials with water. The preferred concentration of the solid content of this slurry is 4—15%, and more preferably, 6—10%. Below 4%, the production efficiency is low, resulting in lowered productivity. Besides, the solid content in the slurry is precipitated, tending to make it impossible to obtain the hardening composition.

However, when the solid content of the slurry becomes above 15%, the thickness of the processed cake becomes irregular, causing difficulty in obtaining a homogeneous hardening composition.

Also, the coefficient of filtration for the slurry must be adjusted to be below 5 cm^4/sec, since such a numeral value is an absolute requirement for implementing production by Hatchek system.

A slurry with the foregoing composition thus obtained is processed by using Hatchek processing machine and stratified into forming the basic composition with appropriate thickness. Through curing this basic composition, the hardening composition is obtained.

It should be apparent from the foregoing description that with the method of the present invention, a high strength hardening composition can be mass-produced even without using asbestos. Furthermore, because the content of the pulp is low and also a fibrillation is done up to a high degree, not only is the hardening composition obtained non-flammable, but also low in rate of water absorption resulting in a smaller reduction in strength upon water absorption. In addition, the fine fibers resulting from the fibrillation improve the adhesion between the layers of the processing products. Hence, the hardening composition is also excellent in resistance to frost damage.

The preferred embodiments of the present invention provide a method for producing inorganic hardened compositions without the use of asbestos which are non-flammable, extremely strong, inexpensive and suitable for mass production.

Examples and Comparisons

The following Examples are given by way of further illustration of the present invention; some examples for comparison are also given.

Examples 1—24 and the Comparison Examples 1—14 of the inorganic hardening composition were prepared from the raw materials shown in Table 1, by using the Hatchek system that uses the Hatchek processing machine. The results of the test conducted by using those samples are also shown in Table 1.

In the table which evaluates the processing efficiency (yield in scooping) and frost damage resistance: ⊙ means satisfactory, and X means unsatisfactory.

Comparison Example 5 was prepared by fibrillating the pulp as in the present invention, but because the concentration of the slurry was excessively low, production was not possible.

Comparison Example 1 is the case using asbestos. Comparison Examples 2—4 were obtained without using asbestos but using the pulp that is below the normal value, i.e., 70°SR, in Schopper freeness.

As shown in Table 1, all of the examples of the present invention were superior to the examples for comparison.

TABLE 1

	Example 1	Example 2	Example 3	Example 4	Example 5
Ordinary Portland cement (kg)	85	87	87	86	94
Fibrillated needle leaf tree pulp (kg)	unbleached 5	—	bleached 2	bleached 2	unbleached 2
Fibrillated broadleaf tree pulp (kg)	—	unbleached 3	unbleached 1	—	—
Pulp below 70°SR in freeness (kg)	—	—	—	2	—
Vinylon Fiber (kg)	—	—	—	—	—
Wollastonite (kg)	—	—	—	—	—
Sepiolite (kg)	—	—	—	—	2
Bentonite (kg)	—	—	—	—	—
Silica Sand (kg)	10	10	10	10	—

TABLE 1 (contd.)

		Example 1	Example 2	Example 3	Example 4	Example 5
Water (ton)		1.15	1.15	1.15	1.15	1.15
Condition for beating pulp		Double Disc refiner 4 cycles	Double Disc refiner 4 cycles	Double Disc refiner 6 cycles	Double Disc refiner 6 cycles	PFI mill 100,000 rotations
Freeness of fibrillated pulp (0.2% water) (°SR)		85	73	85	72	103
Amount of pulp shorter than 177 in fiber length, in fibrillated pulp (%)		4.5	4	7.0	6.8	5.2
Workability in production process (scoop-sieving)		⊙	⊙	⊙	⊙	⊙
Specific gravity of product		1.7	1.7	1.7	1.7	1.7
Bending Strength (kg/cm^2)	In dry state	250	205	210	225	203
	In state with water absorbed	180	155	160	178	148

TABLE 1 (1) (contd.)

	Example 1	Example 2	Example 3	Example 4	Example 5
Inter layer (kg/cm^2) adhesive strength	18	17	14	17	16
(ASTM Ⓐ method 3000 times) Resistance to frost damage	⊙	⊙	⊙	⊙	⊙
Result of non inflammability test	non-flammable	non-flammable	non-flammable	non-flammable	non-flammable
Coefficient of filtration (cm^4/sec)	3.1	2.8	2.9	3.8	4.0
Concentration of slurry (%)	8.2	8.2	8.2	8.2	8.2

1: Fibrillated pulp means that is adjusted to be above 70°SR in Schopper freeness and to contain the pulp with fiber length of less than 177 μ in composition ratio of 10 wt. % of the total pulp content.

Note.
⊙ Excellent
O Good
Δ Fair
X Poor

TABLE 1 (2)

	Example 6	Example 7	Example 8	Example 9	Example 10
Ordinary Portland cement (kg)	92	93	91	86	89.5
Fibrillated needle leaf tree pulp (kg)	4	3	—	3	3
Fibrillated broadleaf tree pulp (kg)	—	—	3	—	—
Pulp below 70°SR in freeness (kg)	—	1	1	1	1
Vinylon Fiber (kg)	—	1	—	1	0.5
Wollastonite (kg)	—	—	5	5	2
Sepiolite (kg)	—	2	—	—	2
Bentonite (kg)	4	—	—	4	2
Silica Sand (kg)	—	—	—	—	—

TABLE 1 (2) (contd.)

		Example 6	Example 7	Example 8	Example 9	Example 10
Water (ton)		1.15	1.15	1.15	1.15	1.15
Condition for beating pulp		Single disc refiner 8 cycles	Double disc refiner 10 cycles	Double disc refiner 10 cycles	Double disc refiner 10 cycles	—
Freeness of fibrillated pulp (0.2% water) (°SR)		85	90	93	90	90
Amount of pulp shorter than 177 in fiber length, in fibrillated pulp (%)		4.3	5.4	6.0	5.8	4.7
Workability in production process (scoop-sieving)		◎	◎	◎	◎	◎
Specific gravity of product		1.7	1.7	1.7	1.7	1.7
Bending Strength (kg/cm^2)	In dry state	234	275	270	295	284
	In state with water absorbed	167	224	201	232	227

TABLE 1 (2) (contd.)

	Example 6	Example 7	Example 8	Example 9	Example 10
Inter layer (kg/cm^2) adhesive strength	19	20	18	21	19
(ASTM Ⓐ method 3000 times) resistance to frost damage	⊙	⊙	⊙	⊙	⊙
Result of non-inflammability test	non-flammable	non-flammable	non-flammable	non-flammable	non-flammable
Coefficient of filtration (cm^4/sec)	1.4	2.5	3.3	1.0	1.2
Concentration of slurry (%)	8.2	8.2	8.2	8.2	8.2

Note

⊙ Excellent
O Good
Δ Fair
X Poor

TABLE 1 (3)

	Comparison Example 1	Comparison Example 2	Comparison Example 3	Comparison Example 4	Comparison Example 5
Ordinary Portland cement (kg)	76	85	82	82	92
Asbestos 6D (kg)	14	—	—	—	—
Fibrillated needle leaf tree pulp (kg)	—	—	—	—	3
Fibrillated broadleaf tree pulp (kg)	—	—	—	—	—
Pulp below 70°SR in freeness (kg)	1	5	8	5	1
Vinylon Fiber (kg)	—	—	—	—	—
Wollastonite (kg)	—	—	—	—	—
Sepiolite (kg)	—	—	—	1	2
Bentonite (kg)	—	—	—	2	2
Silica Sand (kg)	10	10	10	10	—

TABLE 1 (3) (contd.)

		Comparison Example 1	Comparison Example 2	Comparison Example 3	Comparison Example 4	Comparison Example 5
Water (ton)		1.15	1.15	1.15	1.15	1.15
Condition for beating pulp		—	—	—	—	Double disc refiner 6 cycles
Freeness of Fibrillated pulp (0.2% water) (°SR)		—	—	—	—	90
Amount of pulp shorter than 177 in fiber length, in fibrillated pulp (%)		—	—	—	—	5.2
Workability in production process (scoop-sieving)		⊙	x	⊙	x	x
Specific gravity of product		1.7	—	1.6	—	—
Bending Strength (kg/cm²)	In dry state	257	—	242	—	—
	In state with water absorbed	211	—	120	—	—

TABLE 1 (3) (contd.)

	Comparison Example 1	Comparison Example 2	Comparison Example 3	Comparison Example 4	Comparison Example 5
Inter layer (kg/cm^2) adhesive strength	18	—	15	—	—
(ASTM Ⓑ method 3000 times) Resistance to frost damage		—	x	—	—
Result of non-inflammability test	non-combustible	—	semi-non-combustible	—	—
Coefficient of filtration (cm^4/sec)	2.5	1.5	4.8	8.5	4.0
Concentration of Slurry (%)	8.2	8.2	8.2	8.2	2.8

Note
⊙ Excellent
O Good
Δ Fair
X Poor

TABLE 1 (4)		Exp. 11	Exp. 12	Exp. 13	Exp. 14
COMPOSITION	Portland Cement	75	75.3	75.5	78.5
	Virgin Pulp	NUKP 1	NUKP 0.7	NUKP 0.5	NUKP 0.5
	Fibrillated Pulp	NUKP 3	NUKP 3	NUKP 3	NUKP 3
	Organic Fiber	VINYLON 1	VINYLON 1	VINYLON 1	VINYLON 1
	Inorganic Fiber (Wollastonite)	5	5	5	5
	Filler (Sepiolite)				
	Filler (Bentonite)				
	Filler Silica (1 μ in average grain size)	10	10	10	7
	Silica Sand	5	5	5	5
Polymer Floccuant [ppm to ss]		30	30	30	30
Slurry Concentration (%)		8.5	8.4	7.7	8.7
Concentration of Solid Content in Filtrate (%)		1.6	1.3	1.2	1.1
No. of Windings (times)		4	5	4	5
Felt Speed [m/min]		35	35	35	36

TABLE 1 (4) (contd.)	Exp. 11	Exp. 12	Exp. 13	Exp. 14
Pulp Beating Condition & °SR of Fibrillated Pulp	Double Disc Refiner 7 times (73°SR)	Same as Left	Same as Left	Same as Left
Specific Gravity	1.70	1.74	1.78	1.79
Bending Strength in Normal State (vert. dir.)	278	267	274	265
Bending Strength in Saturated State w/Water (vert. dir.)	199	189	211	200
Charpy Impact Strength (vert. dir.)	4.7	4.3	4.9	4.1
Interlayer Adhesive Strength (kg/cm²)	17.5	16.8	17.7	18.0
Test Result for Non-Flammability	OK	OK	OK	OK
Anti-Frost Melt Cycle (ASTM-A) Test	⊙	⊙	⊙	⊙
Dimensional Variation Rate	0.21	0.19	0.18	0.23

TABLE 1 (4) (contd.)		Exp. 15	Exp. 16	Exp. 17	Exp. 18
COMPOSITION	Portland Cement	74.3	78.3	76.3	74.5
	Virgin Pulp	NUKP 0.7	NUKP 0.7	NUKP 0.7	NUKP 0.5
	Fibrillated Pulp	NUKP 3	NUKP 3	NUKP 3	NUKP 4
	Organic Fiber	VINYLON 2	VINYLON 1	VINYLON 1	VINYLON 1
	Inorganic Fiber (Wollastonite)	5	5	5	5
	Filler (Sepiolite)		2		
	Filler (Bentonite)			4	
	Filler Silica (1 μ in average grain size)	10			10
	Silica Sand	5	10	10	5
Polymer Flocculant [ppm to ss]		30	30	30	30
Slurry Concentration (%)		7.5	7.7	7.8	8.2
Concentration of Solid Content in Filtrate (%)		1.4	1.3	1.0	0.9
No. of Windings (times)		4	4	4	4
Felt Speed [m/min]		35	35	35	34

TABLE 1 (4) (contd.)	Exp. 15	Exp. 16	Exp. 17	Exp. 18
Pulp Beating Condition & °SR of Fibrillated Pulp	Same as Left	Same as Left	Double Disc Refiner 10 times (80°SR)	Same as Left
Specific Gravity	1.72	1.76	1.74	1.70
Bending Strength in Normal State (vert. dir.)	231	284	265	260
Bending Strength in Saturated State w/Water (vert. dir.)	168	199	182	175
Charpy Impact Strength (vert. dir.)	5.7	4.4	4.0	4.5
Interlayer Adhesive Strength (kg/cm^2)	13.4	15.8	15.0	16.8
Test Result for Non-Flammability	OK	OK	OK	OK
Anti-Frost Melt Cycle (ASTM-A) Test	⊙	⊙	⊙	⊙
Dimensional Variation Rate	0.17	0.20	0.21	0.17

TABLE 1 (4) (contd.)		Exp. 19	Exp. 20	Exp. 21	Exp. 22
COMPOSITION	Portland Cement	78.3	78.0	76.0	74.0
	Virgin Pulp	NUKP 0.7	NUKP 1	NUKP 1	NUKP 1
	Fibrillated Pulp	NUKP 3	NUKP 3	NUKP 3	NUKP 4
	Organic Fiber	VINYLON 1	ACRYL 1	ACRYL 1	VINYLON 1
	Inorganic Fiber (Wollastonite)	5	5	5	5
	Filler (Sepiolite)		2	2	
	Filler (Bentonite)			2	
	Filler Silica (1 μ in average grain size)	7			10
	Silica Sand	5	10	10	5
Polymer Flocculant [ppm to ss]		30	30	30	30
Slurry Concentration (%)		8.0	7.5	8.5	7.6
Concentration of Solid Content in Filtrate (%)		1.4	1.7	1.6	1.2
No. of Windings (times)		4	4	4	4
Felt Speed [m/min]		36	37	35	36

TABLE 1 (4) (contd.)	Exp. 19	Exp. 20	Exp. 21	Exp. 22
Pulp Beating Condition & °SR of Fibrillated Pulp	Double Disc Refiner 7 times (73°SR)	Same as Left	Same as Left	Same as Left
Specific Gravity	1.77	1.68	1.71	1.65
Bending Strength in Normal State (vert. dir.)	277	248	265	261
Bending Strength in Saturated State w/Water (vert. dir.)	203	188	200	191
Charpy Impact Strength (vert. dir.)	3.7	2.8	3.0	2.9
Interlayer Adhesive Strength (kg/cm^2)	17.4	15.2	18.2	14.4
Test Result for Non-Flammability	OK	OK	OK	OK
Anti-Frost Melt Cycle (ASTM-A) Test	⊙	⊙	⊙	⊙
Dimensional Variation Rate	0.19	0.23	0.24	0.18

TABLE 1 (4) (contd.)		Comparison Exp. 6	Comparison Exp. 7	Comparison Exp. 8	Comparison Exp. 9
COMPOSITION	Portland Cement	76.0	75.0	74.0	73.0
	Virgin Pulp			NUKP 2	NUKP 3
	Fibrillated Pulp	NUKP 3	NUKP 4	NUKP 3	NUKP 3
	Organic Fiber	VINYLON 1	VINYLON 1	VINYLON 1	VINYLON 1
	Inorganic Fiber (Wollastonite)	5	5	5	5
	Filler (Sepiolite)				
	Filler (Bentonite)				
	Filler Silica (1 μ in average grain size)	10	10	10	10
	Silica Sand	5	5	5	5
Polymer Flocculant [ppm to ss]		50	50	50	30
Slurry Concentration (%)		9.2	8.4	9.0	7.9
Concentration of Solid Content in Filtrate (%)		1.0	1.0	2.4	3.3
No. of Windings (times)		5	4	4	4
Felt Speed [m/min]		36	35	31	35

TABLE 1 (4) (contd.)	Comparison Exp. 6	Comparison Exp. 7	Comparison Exp. 8	Comparison Exp. 9
Pulp Beating Condition & °SR of Fibrillated Pulp	Same as Left	Same as Left	Same as Left	Same as Left
Specific Gravity	1.60	1.48	1.48	1.44
Bending Strength in Normal State (vert. dir.)	220	214	228	230
Bending Strength in Saturated State w/Water (vert. dir.)	154	130	150	167
Charpy Impact Strength (vert. dir.)	2.2	2.8	2.9	2.4
Interlayer Adhesive Strength (kg/cm²)	11.5	11.6	12.4	10.9
Test Result for Non-Flammability	OK	OK	FAILED	FAILED
Anti-Frost Melt Cycle (ASTM-A) Test	⊚	⊚	○	Δ
Dimensional Variation Rate	0.16	0.29	0.32	0.27

TABLE 1 (4) (contd.)		Comparison Exp. 10	Comparison Exp. 11	Comparison Exp. 12
COMPOSITION	Portland Cement	63.0	65.0	75.8
	Virgin Pulp	NUKP 1		
	Fibrillated Pulp	3	NUKP 4	NUKP 4
	Organic Fiber	VINYLON 1	VINYLON 1	VINYLON 0.2
	Inorganic Fiber (Wollastonite)	18	5	5
	Filler (Sepiolite)			
	Filler (Bentonite)			
	Filler Silica (1 μ in average grain size)	10	20	10
	Silica Sand	5	5	5
Polymer Flocculant [ppm to ss]		30	30	30
Slurry Concentration (%)		8.5	7.9	8.2
Concentration of Solid Content in Filtrate (%)		1.5	1.6	1.2
No. of Windings (times)		4	4	4
Felt Speed [m/min]		34	33	36

TABLE 1 (4) (contd.)	Comparison Exp. 10	Comparison Exp. 11	Comparison Exp. 12
Pulp Beating Condition & °SR of Fibrillated Pulp	Same as Left	Same as Left	Same as Left
Specific Gravity	1.49	1.63	1.77
Bending Strength in Normal State (vert. dir.)	211	225	175
Bending Strength in Saturated State w/Water (vert. dir.)	144	153	120
Charpy Impact Strength (vert. dir.)	3.3	3.0	1.9
Interlayer Adhesive Strength (kg/cm²)	7.8	13.2	10.5
Test Result for Non-Flammability	OK	OK	OK
Anti-Frost Melt Cycle (ASTM-A) Test	⊙	⊙	○
Dimensional Variation Rate	0.26	0.27	0.28

TABLE 1 (4) (contd.)		Exp. 23	Comp. Exp. 13	Exp. 24	Comp. Exp. 14
COMPOSITION	Ordinary Portland Cement	80 (kg)	80 (kg)	80 (kg)	80 (kg)
	Needleleaf Tree Pulp	4	4	4	4
	Vinylon Fiber	1	1	1	1
	Wollanstonite	5	5	5	5
	Silica Sand	10	10	10	10
	Water	1.15 (ton)	1.15 (ton)	1.15 (ton)	1.15 (ton)
CONDITIONS FOR BEATING PULP	Concentration of Beaten Pulp (%)	4.5	4.5	4.5	4.5
	Blade Thickness of Double Disc Refiner (mm)	5	4	5	4
	Interblade Distance of Double Disc Refiner	5	4	5	4
	Number of Treatments by Double Disc Refiner (Time)	5	6	6	10

TABLE 1 (4) (contd.)		Exp. 23	Comp. Exp. 13	Exp. 24	Comp. Exp. 14
BEATEN PULP	Freeness (0.2% aqueous solution) (SR°)	60	60	75	75
	Amount of Pulp with Fiber Length of Less than 177 μ (Ratio to Total Amount of Pulp) (%)	7	20	9	35
	Amount of Pulp with Fiber Length of More than 590 μ (Ratio to Total Amount of Pulp) (%)	70	50	65	35

Examples and Comparison Examples
(When pulp is fibrillated with getting cut)

TABLE 1 (4) (contd.)			Exp. 23	Comp. Exp. 13
PROCESSING CONDITIONS	Coefficient of Filtration (cm^4/sec)		3.8	3.8
	Concentration of Slurry (%)		8.2	8.2
	Workability of Processing		⊙	⊙
PHYSICAL PROPERTIES OF BOARD	Specific Gravity of Product		1.7	1.7
	Bending Strength	When Dried (kg/cm^2)	295	208
		When Absorbed Water (kg/cm^2)	218	115
	Interlayer Adhesive Strength (kg/cm)		17	10
	Frost Damage Resistance (ASTM) (A:E 300 times)		No Abnormality is shown after 300 times	Partially Separated by 100 times
	Test Result for Non-Flammability		Non-Flammable	Non-Flammable

Note
⊙ Excellent
O Good
Δ Fair
X Poor

TABLE 1 (4) (contd.)			Exp. 24	Comp. Exp. 14
PROCESSING CONDITIONS	Coefficient of Filtration (cm^4/sec)		2.5	2.5
	Concentration of Slurry (%)		8.2	8.2
	Workability of Processing		⊙	⊙
PHYSICAL PROPERTIES OF BOARD	Specific Gravity of Product		1.7	1.7
	Bending Strength	When Dried (kg/cm^2)	280	183
		When Absorbed Water (kg/cm^2)	214	106
	Interlayer Adhesive Strength (kg/cm)		16	11
	Frost Damage Resistance (ASTM) (A:E 300 times)		No Abnormality is shown after 300 times	Partially Separated by 100 times
	Test Result for Non-Flammability		Non-Flammable	Non-Flammable

Note:
⊙ Excellent
O Good
Δ Fair
X Poor

CLAIMS

1. A method for producing an inorganic hardened composition including the step of curing a composition which is obtained from a slurry containing cement by using a production process such as for example scooping-sieving, said slurry comprising; pulp accounting for 1—5 wt. % (% by weight) of the total solid content of the slurry, 10 wt. % or less of said pulp being of 177 or shorter in fiber length and being adjusted with its Schopper freeness at 70°SR or higher through fibrillation; and filler having a swelling degree of more than three times and reinforcing fibers upon necessity, in order to adjust the concentration to be 4—15 wt. % and the filtration coefficient to be below 5 cm^4/sec for said slurry.

2. A method according to Claim 1, wherein said pulp is bleached or unbleached pulp of needleleaf trees and/or broadleaf trees.

3. A method according to Claim 1 or 2, wherein a part of said pulp has Schopper freeness of below 70°SR.

4. A method according to any one of Claims 1, 2 or 3, wherein said filler is sepiolite and/or bentonite, content of said filler being 1—5 wt. % of the total solid content.

5. A method according to Claim 1 or Claim 2, wherein said reinforcing fiber is vinylon fiber of 5—50 μ in thickness and 3—10 mm in length, said reinforcing fiber being 0.3—2 wt. % to the total solid content.

6. A method according to Claim 3, or Claim 4, wherein said reinforcing fiber is the vinylon fiber of 5—50 μ in thickness and 3—10 mm in length, the content ratio of said reinforcing fiber being 0.3—2 wt. % of the total solid content.

7. A method according to any one of Claims 1 to 6, wherein said reinforcing fiber is wollastonite accounting or 2—15 wt. % of the total solid content.

8. A method for producing an inorganic hardened composition including the step of curing a composition which is obtained from a slurry containing cement using a production method such as, for example, scoop-sieving, said slurry comprising: wood pulp accounting for 1—5 wt. % of the total solid content of the slurry, 60 wt. % or more of said wood pulp being of 590 μ (28 mesh) or longer in fiber length and adjusted with its Schopper freeness at 40—95°SR; unbeaten wood pulp of below 40°SR in Schopper freeness, said unbeaten wood pulp accounting for 0.5—1.0 wt. % of the total solid content of the slurry; and filler and reinforcing fibers, if necessary, to be compounded in order to adjust the concentration to be 4—15 wt. % and the filtration coefficient to be below 5 cm^4/sec for said slurry.

9. A method according to Claim 8, wherein the pulp is either bleached or unbleached wood pulp from needleleaf trees and/or broadleaf trees.

10. A method according to Claim 8 or 9, wherein the filler is sepiolite and/or bentonite, and/or crystal or noncrystal silica of below 5 μ in average grain size, said filler being 1—10 wt. % to the total solid content.

11. A method according to any one of Claims 8, 9 or 10, wherein the reinforcing fiber is vinylon and/or acrylic fiber of 5—50 μ in thickness and 3—10 mm in length, said reinforcing fiber being 0.3—2 wt. % to the total solid content.

12. A method according to any one of Claims 8 to 11, wherein the reinforcing fiber is wollastonite accounting for 2—15 wt. % of the total solid content.

13. A method according to any one of Claims 8 to 12, wherein the reinforcing fiber is vinylon and/or acrylic fiber having a thickness of 5—50 μ and a length of 3—10 mm, and is 0.3—2 wt. % to the total solid components, the surface of said reinforcing fiber being partially formed uneven.

14. A method for producing an inorganic hardened composition substantially as hereinbefore described in any one of Examples 1 to 24.

15. A method for producing an inorganic hardened composition as claimed in Claim 1 substantially as hereinbefore described.

16. An inorganic hardened composition whenever made by the method of any foregoing claims.

17. An inorganic hardened composition which has been made by curing a composition which has been obtained from a cement-containing slurry including pulp accounting for 1—5 wt. % (% by weight) of the total solid content of the slurry, 10 wt. % or less of said pulp being of 177 μ or shorter in fiber length and being adjusted with its Schopper freeness at 70°SR or higher through fibrillation; and filler having a swelling degree of more than three times and reinforcing fibers upon necessity, in order to adjust the concentration to be 4—15 wt. % and the filtration coefficient to be below 5 cm^4/sec for said slurry.

18. An inorganic hardened composition which has been made by curing a composition which has been obtained from a cement-containing slurry including wood pulp accounting for 1—5 wt. % of the total solid content of the slurry, 60 wt. % or more of said wood pulp being of 590 μ (28 mesh) or longer in fiber length and adjusted with its Schopper freeness at 40—95°SR; unbeaten wood pulp of below 40°SR in Schopper freeness, said unbeaten wood pulp accounting for 0.5—1.0 wt. % of the total solid

content of the slurry; and filler and reinforcing fibers, if necessary, to be compounded in order to adjust the concentration to be 4—15 wt. % and the filtration coefficient to be below 5 cm^4/sec for said slurry.

19. A building material (e.g. a board, brick, tile etc.) including an inorganic hardened composition as claimed in any one of Claims 16 to 18.

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